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Pinhole resistance of flexibles

Part I: New methods measure puncture, flexing and abrasion in films and laminates

By K.H. Hu and J.B. Breyer

No matter how small pinholes are, they can have detrimental effects on package performance by allowing transport of water vapor and oxygen in or out of the package. This was conclusively proved in our previous work (1)*. Under certain conditions, pinholes will allow entry of bacteria into a package, thus causing spoilage of food products. In this article, pinholes are defined as small but complete breakthrough openings in a flexible-packaging material—in a laminate, through all components; in a single-layered web, through the film.

Major causes of pinhole formation in a flexible package can be attributed to three modes of mechanical damage—puncture, flexing action and abrasion. For example, pinholes due to puncture can occur when products with sharp corners such as dehydrated or freeze-dried food items are enclosed in a flexible package, especially when a vacuum is applied in the course of packaging. Pinholes due to flexing action can occur when the fold or seal area of a package is flexed during transportation and handling. Pinholes due to abrasion can occur when high points or curved areas of a package rub against another package or against the exterior container.

As soon as a pinhole is formed, the package

*Numbers in parentheses indicate References appended.

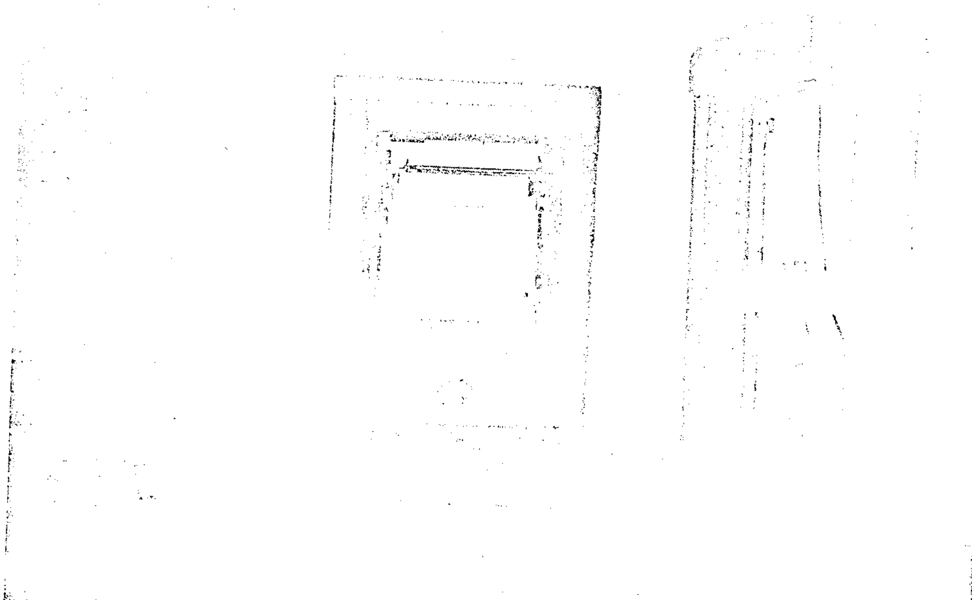

Figure 1. Instron (right) and integrator (center) for measuring puncture forces. Note test cell and needle on Instron.

can be regarded as having lost one of its main functions. Therefore, an understanding of resistance of materials to pinhole formation resulting from abuse can be of vital importance to maintaining adequate levels of package performance. Furthermore, pinhole formation can be used as a design criterion in defining material resistance to mechanical damages with the proper evaluation of these three testing factors.

Measuring methods

Methods employed in our studies are based on the consideration that pinhole formation in a flexible package represents damage resulting from puncture, flexing action or abrasion. Therefore, in measuring material resistance to these damage modes, pinhole formation can be conveniently used as one of the main criteria. Most existing test methods for polymeric materials (2) do not simulate the type of pinhole anticipated in flexible-packaging materials. Therefore, they were not employed in our testing. During the course of our work, two existing test methods were modified and one new one was developed to meet our need for tests that produced pinhole formation by abrasion, flexing action and puncture. They are as follows:

Puncture resistance. Methods for measuring puncture resistance of polymeric materials have been described by Furno et al (3) and Lynch (4). The former used a 0.25-in. steel rod as a plunger, while the latter used a rounded, cylindrical penetrator of 0.19 in. diameter. Based on our observations, the pinholes that develop in flexible packages are much smaller in size than either of these two penetrators. Such pinholes are on the order of 0.01 in. diameter and sometimes less (1).

An ASTM penetration needle of 0.006 in. diameter (0.14 to 0.16 mm.) at the tip of the needle was used as our puncturing device (5). The machine is an Instron Tester with compression-load cell attached. An integrator was hooked up with the Instron, as shown in Fig. 1, so that both maximum force and total energy absorbed in puncturing could be measured on all types of flexible materials.

The penetration needle was held upright in the compression cell. The flexible material was placed in a holder that consists of a modified Thwing-Albert Vapometer cup provided with two circular,

The authors: Messrs. Hu and Breyer are with the Engineering Sciences Div., General Equipment & Packaging Laboratory, U.S. Army Natick Laboratories, Natick, Mass.

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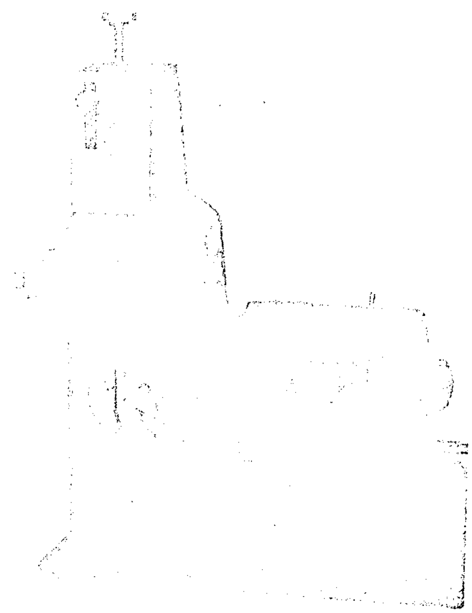

Figure 2. Folding endurance tester for gauging flexing action. Sample is clamped at left.

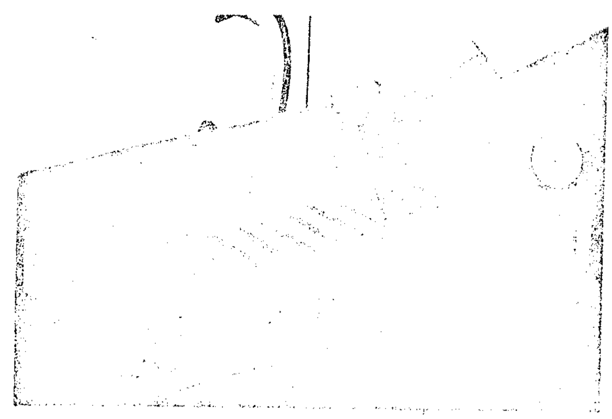

Figure 3. Vacuum box for detecting pinholes by transmission of dye through flexed samples.

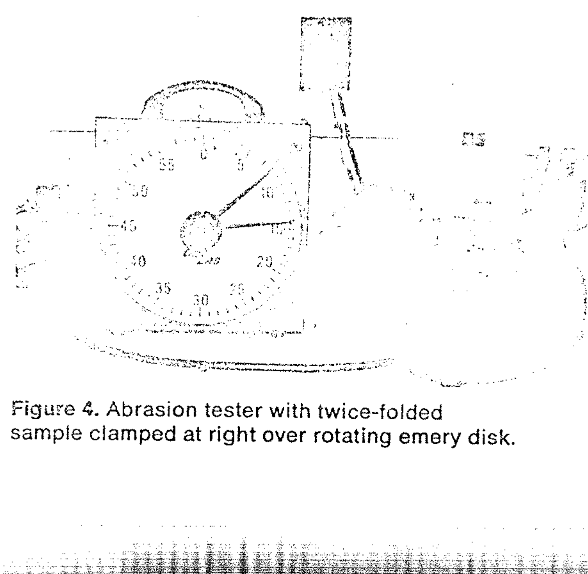

Figure 4. Abrasion tester with twice-folded sample clamped at right over rotating emery disk.

rigid, metal disks having a 0.5-in.-diameter hole cut in the center of each. A rubber O-ring was placed between the disks along with the sample of flexible material, and the assembly was then clamped into the Vapometer cup. The completely assembled holder was attached to the movable bar of the Instron machine directly above the compression cell (right center in Fig. 1).

The Instron machine was set up as follows:

a. CB compression cell: range 100 to 2,000 gm.

b. Jaw speed: 0.2 in. per min.

c. Chart speed: 5.0 in. per min.

Ten readings for each material were taken and an average of these readings was made.

Flexing resistance. An MIT Folding Endurance Tester (Fig. 2) and a specially designed vacuum box (Fig. 3) were employed for this test (6). The test sample was first given a predetermined number of flexes in the tester, then was removed and mounted on the vacuum box to test for the presence of pinholes.

The sample to be tested was cut 5.5 by 1.2 in. and folded in half, lengthwise, with the heat-scalable surface inside the fold. It was then placed in the tester jaws with the folded edge facing the body of the machine. The tension of the jaw was set at 0.5 kg. The speed was set at 180 cycles per min.

After the predetermined flexing cycle was completed, the sample was unfolded and taped to the top of the vacuum box with a strip of filter paper underneath. A drop of dye solution was placed at the flexing point of the test sample. A 23.5-in. vacuum was drawn at the vacuum box. The appearance of dye on the filter paper indicated that flexing had caused a pinhole in the test sample. Ten readings were taken at each number of cycles selected for testing. When nine or 10 readings out of a total of 10 showed dye penetration, this number of cycles was then taken as the one that causes pinhole formation in the particular material that was being tested.

Abrasion resistance. A new abrasion tester was developed and used in our test work (7). A 3-by-3-in. section was cut from the test sample. It was folded diagonally once, forming a triangle. It was folded again in the middle of the first fold, forming a second triangle half the size of the first triangle. The twice-folded sample was then inserted into a holder in such a manner that its tip was placed against an abrasive material, as shown at right in Fig. 4.

The abrasive material, emery polishing paper of grit No. 4/0, was mounted on a turntable that was spun at 50 rpm. The test sample was removed from the folder after a predetermined number of revolutions was completed. A drop of dye solution was placed inside the sample fold and a piece of filter paper was used to touch the tip of the fold on the outside. Appearance of dye on the filter paper indicated that a pinhole had developed. Ten readings at each number of revolutions for testing were taken. When nine or 10 readings out of a total of 10 showed dye penetration, this number of revolutions was then taken as the one that caused pinhole formation.

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The authors would like to thank Messrs. Laurence Kooklin and John Kessler, who conducted many tests and obtained a great body of data, and are grateful to Mr. Earl Steeves for first elucidating the fact that *the puncture resistance of a laminate is usually less than the sum of those values obtained from individual components* can be explained from an analysis of strength of materials. The authors also wish to thank Dr. Leslie McClaine, Dr. Rauno Lampi and Mr. Gerald Schultz for reviewing this manuscript.

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Next month, in a concluding Part II to this article, the results of these tests and the methods used will be discussed in full. Among the interesting conclusions is the fact that most laminates are strong in one or two properties, but weak in others. This necessitates a trade-off in properties, depending on the specific packaging application for which the material is designed.

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TECHNICAL/ENGINEERING

Pinhole resistance of flexibles

Part II: Results and discussion of new film tests to determine puncture, flexing and abrasion

By K.H. Hu and J.B. Breyer

Three new tests have been devised to measure puncture, flexing action and abrasion—the key factors in pinhole formation in flexible materials. These techniques were described last month (MP, Dec. '70, p. 46) Now follows, in this second and concluding part, an analysis of the testing techniques.

Effect of material thickness. Results of tests of polytrifluoromonochloroethylene, polyethylene terephthalate and polyamide films all show that an increase in thickness of flexible material increases its puncture and abrasion resistance but decreases its flexure resistance. Typical are the results with polytrifluoromonochloroethylene film, illustrated in Fig. 5. There, the maximum force required for puncturing varies approximately linearly with material thickness, whereas the total energy absorbed, cycles of flexing and revolutions of abrasion, do not follow this simple relationship.

In other words, for flexible packaging, increasing material thickness does not always improve package performance. Performance will depend on which mode of damage plays the most important role. If puncture or abrasion damage is the main contributing factor, an increase in material thickness can improve package protection. But, if flexing action is the main factor, an increase in thickness will result in the opposite effect.

Effects of lamination. Modern flexible-packaging materials usually consist of two to four layers (films and metal foil) adhered together. The adhesive layer employed in the laminates used in our testing was very thin, about 0.5 lbs. per 1,000 sq. ft., and in a broad sense belongs to the polyester-epoxy type.

Resistance to puncture. When several components (or films) are brought together in a lamination, thickness is increased and, therefore, puncture resistance is increased. When we examine the relationship between the puncture resistance of a laminate and that of the components that make up the laminate, the former is usually less than the sum of the latter. In other words, we usually do not get the total amount of puncture resistance expected on the basis of individual components in the laminate.

This is indicated in the results shown in Table I. There, for the first laminate, we determined the maximum force required to puncture the laminate from "inside" was 178 gm., which is smaller than the sum of the components that was determined separately as 205 gm. This effect was also observed for the total energy absorbed in puncturing.

Because there were adhesives used in the laminates and, consequently, a possibility of synergetic action among materials, one might expect the force required to puncture a laminate to be greater than the sum of the components determined individually. Our results show the contrary. An explanation lies in the analysis of strength of materials.

It is well known that in an axially loaded member of two or more materials of the same length, the unit stress in each is directly proportional to their (a) modulus of elasticity. It is also obvious that, other things being equal, failure resistance of a component is directly proportional to (b) the breaking strength of the material involved. Because of these two determining factors, there are several possible interactions.

The component with high modulus of elasticity may have low breaking strength, or the component with low modulus of elasticity may have a high breaking strength in relationship to other components. One typical example is shown in Fig. 6, in which F_1 and F_2 are forces required to cause failure of components 1 and 2, determined individually; P_1 and P_2 are forces supported by components 1 and 2, when they are in a composite under Load P_t. In equilibrum:

$$P_t = P_1 + P_2$$

When load P_t is increased, component 1 (high modulus of elasticity, but low breaking strength), as shown in Fig. 6, carries more of the load until $P_1 = F_1$ (component 1 fails). Then, $P_2 = P_t$ (component 2 must now support the entire load). When P_t is increased further to $P_t = F_2$, composite failure takes place. Therefore, at the composite failure:

$$P_t = F_2 < F_2 + F_1$$

The above derivation implies that the two components in a composite break at two different points in the loading process, thus contributing to a lower force than the sum of the two components determined individually.

In spite of several possible combinations of

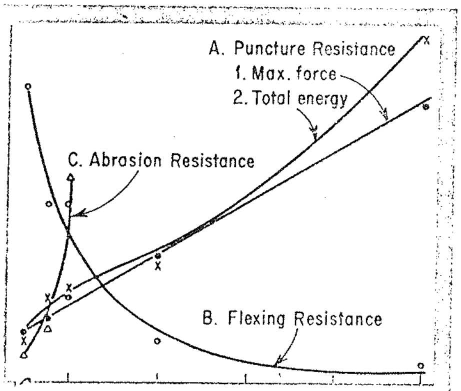

Figure 5. Effects of material thickness on pinhole formation.

Figure 6. Force displacement chart showing two peaks of failure for two laminates.

modulus of elasticity and breaking strength of one component in relationship to other components, the force required to cause failure of a laminate is always less than, or at the most equal to, the sum of the components determined individually. This behavior of the axially loaded member coincides with our observations for the puncture of laminates, where two components in a laminate can actually fail at two different points, as indicated by two peaks in the force displacement chart, shown in Fig. 7.

It also should be mentioned that there are notable differences in forces and energies between punctures initiated from the "outside" or from the "inside" of a laminate, as shown in Table I. But there is no general rule as to which direction of puncturing requires greater force and energy. It seems to depend on the thickness and physical characteristic of the components that make up the laminate.

Resistance to flexing action. An increase in materials thickness, based on our three test films, results in a decrease of resistance to flexing action. This observation also applies to the effect of lamination. In laminations, the increased thickness, as would be expected, decreases drastically resistance to flexing action, as shown in Table II.

Resistance to abrasion. Laminating increases greatly the abrasion resistance of films, as shown in Table III.

When puncture-flexing action and abrasion resistances are all taken into consideration, many laminates under study show up strong in one or two resistance properties, but weak in others. Typical examples are shown in Table IV. There are very few laminates that can be considered strong in resistance to all three damage modes. For practical applications, compromises have to

Table I: Puncture resistance of a laminate and of its individual components

Materials used in laminate construction	Components determined separately		Laminate determination			
			max. force, gm.		total energy, in.-gm.	
	max. force, gm.	total energy, in.-gm.	from "inside"*	from "outside"*	from "inside"*	from "outside"*
0.5-mil polyester	106	1.47				
0.35-mil aluminum foil	23	0.13				
3.0-mil HDPE	76	2.10				
Total	205	3.70	178	203	2.81	2.73
2.0-mil polyester	331	4.36				
0.35-mil aluminum foil	23	0.13				
2.0-mil vinyl	90	4.15				
Total	444	8.64	422	315	6.96	4.41

*"Inside" is heat-seal side; "outside" is polyester side.

Table II: Effects of lamination on flexing action

Laminate construction	Cycles of flexing action to cause pinhole formation		
	Three components determined separately	Two-component laminate (a and c)	Three-component laminate (a, b and c)
(a) 0.5-mil polyester	10,000	3,000	700
(b) 0.35-mil aluminum foil	23		
(c) 3.0-mil HDPE	5,000		

Table III: Effects of lamination on abrasion resistance

Laminate construction	Revolutions of abrasion to cause pinhole formation	
	Three components determined separately	Three-component laminate (a, b and c)
(a) 2.0-mil polyester	50	900
(b) 0.35-mil aluminum foil	5	
(c) 2.0-mil vinyl	10	

Table IV: Laminate differences in resistance to damage modes

	Puncture resistance from "inside"*, gm.	Flexing resistance, cycles to pinhole formation	Abrasion resistance, revolutions to pinhole formation
2.0-mil polyester/0.35-mil aluminum foil/2-mil vinyl*	518	3,000	900
0.5-mil polyester/0.35-mil aluminum foil/3-mil HDPE	178	700	1,000
0.5-mil polyester/0.35-mil aluminum foil/3-mil polyolefin	148	10,000>	800

*"Inside" is heat-seal side. Note: Construction No. 1 is too stiff for flexible packages and is used here only for purposes of comparison.

be made, and the mode of damage most important in the packaging application should be ascertained before an intelligent choice of laminate material can be made.

Variations in testing speeds

Consideration was given to use of different speeds in procedures to determine if speeds other than the one used in the adopted test procedure would change the relative position of materials with respect to their resistance to pinhole formation.

With our experimental setup for determining resistance to puncture, we found that the maximum jaw speed that could be employed with the Instron and still get accurate readings was 1 in. per min. At a speed of 2 in. per min., the laminate usually broke too quickly to get accurate readings. In general, our results indicated that the maximum force and energy required for puncture increased when the speed of puncture was increased. However, when speed was increased from 0.2 to 1 in. per min., the relative position of laminates did not change.

Firm conclusions were not possible as to the effects of flexing speed on flexure resistance. Results from two speeds of flexing action (132 cycles per min. and 180 cycles per min.) showed no change in the relative position of materials under study.

As far as measuring abrasion resistance is concerned, increase in the turntable speed of our abraser increases the number of samples with pinhole formation, but in general, the relative position of materials with respect to abrasion resistance did not change.

Conclusions

Pinhole formation in flexible-packaging materials resulting from puncture, flexing action and abrasion was studied and identified. Methods and appropriate instrumentation were either adopted or developed to closely simulate and measure these three damage modes under controlled laboratory conditions.

Laboratory results have established several general rules governing flexible packaging materials:

(a) Increase in thickness of a flexible material increases its puncture and abrasion resistance, but decreases its flexing resistance.

(b) When several components (or films) are laminated: puncture resistance of the laminate is usually less than the sum of the individual components; flexing resistance is reduced drastically, and abrasion resistance increases dramatically.

(c) Changes in test speeds do not appear to change the relative position of materials with respect to pinhole formation.

(d) Many laminates show up strong in one or two resistance properties, but weak in others. In practical packaging situations, compromises have to be made. The predominant damaging factor must be ascertained for a specific application so that an intelligent choice of materials, or combination of materials, can be made. □